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Improved model for capillary absorption in cementitious materials: Progress over the fourth root of time

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ABSTRACT

Sorptivity is broadly used for characterising the pore connectivity of cementitious materials, with applications in design for durability. A water sorptivity coefficient (WSC) is typically obtained from the ratio between the amount of absorbed water and $t^{0.5}$. This relationship is however not linear for cementitious materials, and conventions are needed for the computation. Variable criteria in the literature complicate the comparison of WSCs. This paper proposes a new approach for describing the entire absorption process. We substantiate the hygroscopicity of calcium silicate hydrates and the effect of swelling during the process as the main causes for the anomalous capillary absorption by cementitious materials. We present a theoretical model with a single descriptive coefficient of capillary absorption progressing linearly with $t^{0.25}$. The model fits remarkably well to experimental data, and it solves the problem of lack of linearity with $t^{0.5}$. A full description of the transport process is then offered.

1. Introduction

Either performance-based or prescriptive perspectives are possible for designing concrete that will be exposed to aggressive environments. Performance-based design for durability demands sustained relationships between service conditions and parameters of concrete with values that can be defined to comply with a certain lifespan of the structure, such as the thickness of cover concrete and its properties. Prescriptive design for durability lies on the conformity of values for parameters of concrete such as the compressive strength, water/cementitious materials ratio (w/cm), minimum binder content, and others.

Pore sizes and connectivity in concrete defines transport properties. When unsaturated concrete is in direct contact with water, capillary absorption occurs due to the action of forces of adhesion of water molecules to the pore walls in concrete. This transport process may promote the ingress of aggressive agents into concrete and hence affect durability.

The mechanism of the water ingress process is not the only reason to use the capillary absorption rate in the design of durable concrete. The literature offers plenty of examples of relationships between the water sorptivity coefficient (WSC) (generally expressed in units of mass/area/time$^{0.5}$) and other durability and transport properties such as drying rate (DR) [1], chloride diffusion [2], carbonation [3], water penetration under pressure [2,4,5], resistance against freeze and thaw [6–8]. Consequently, the sorptivity is not only used to quantify this process occurring in concrete under service but also as a descriptor of other transport properties of concrete related to each environmental class in particular. Here, a low rate of water absorption indicates satisfactory properties to ensure an acceptable lifespan of a concrete structure. The WSC of concrete could be applied as a durability index for a performance-based approach to achieve durability of reinforced concrete structures regarding carbonation, sulphate attack, freeze and thaw, chloride penetration. However, it faces significant limitations as a design tool for durability given that the quantification of the WSC is still unable to provide a reliable estimation of transport properties of concrete. Moreover, its application requires the adoption of conventions for defining the proper procedure for obtaining the WSC due to a lack of linearity with $t^{0.5}$. Most of the limitations are related to the computational method for obtaining the parameter.

2. The experimental method

The experimental method involves a preconditioning of the sample that includes drying and waterproofing of lateral faces. Then, the bottom flat face of the sample is put into contact with water (only a few

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millimetres), avoiding immersion. The sample gains weight due to water uptake, which is expressed per unit surface area and contrasted with the time elapsed from the start of the contact.

The dependence of WSC on the moisture content of the samples [9–11] has been a significant obstacle for standardisation of the test. In this sense, drying at 105 °C is usually considered a very reproducible preconditioning, but the alteration of the pore structure leads to unrealistically high WSC values due to deterioration of hydration products [12]. Drying at 105 °C induces interconnected microcracking, with an average crack width of 0.5–10 μm [13]. Some microcracking with drying at 50 °C is also reported in [13] but in a much more limited extent. As a consequence, the WSC of concrete can increase by 100% when drying temperature is increased from 50 °C to 105 °C [13]. Drying at low temperature is harder to reproduce, but a necessary procedure for obtaining a realistic WSC. The RILEM TC 116-PCD recommended a preconditioning of concrete to produce a saturation degree equivalent to the one that develops when it is in equilibrium with an environment at RH = 75% [14]. With this aim, the first requirement is the determination of the water loss of saturated concrete when dried in an atmosphere at RH = 75%, and this relative water loss is replicated in the samples by following an accelerated procedure using an oven at 50 °C. The preconditioning is finalised with a moisture redistribution phase. The Spanish standard [15] adopted this procedure. The Argentine standard [16] includes a simplified approach that considers constant weight at 50 °C with variations lower than 0.1% in weight over 24 h, but which can be very sensitive to the oven type and its features [17]. The ASTM standard [18] attempts to assure repeatable preconditioning by conditioning the sample at a certain internal relative humidity by oven drying at 50 °C and subsequent wrapping and storage for homogenization. This relative humidity corresponds to a certain moisture content depending on the pore size distribution. Other European standards for cementitious materials reduce the drying temperature to 40 °C [19–21], or dry in an atmosphere at 20 °C and 50% RH [22,23] or 65% RH [24], to assure minimal damage is caused to the sample. However, these procedures might decrease reproducibility even more, and some of them are extremely time consuming.

According to previous investigations [9,25,26], different drying regimes will have a different impact on concrete microstructure and therefore will alter in a variety of ways the results of the absorption test. Dias [9] claims that the desorption ratio explains to a large extent the variations in sorptivity. This desorption ratio is defined as the weight loss from the saturated condition (i.e. from the end of curing by water immersion) to the start of the sorptivity test, divided by the weight of the saturated specimen. Hence, whatever the concrete grade, specimen preconditioning, specimen size, or specimen coating, the ensuing sorptivity may be linked to the desorption ratio [1]. The energy applied for drying the samples also affects the relationship between the water uptake and $t^{0.5}$ [27], where the application of 105 °C for drying favours linearity with $t^{0.5}$.

3. The origin and defects of the typical analysis of capillary absorption as a function of $t^{0.5}$

The WSC is computed as the slope of the fitting line to the amount of water uptake per unit area as a function of the square root of the time elapsed along which concrete was in contact with water. This approach is applied to analyse capillary absorption in most of porous materials.

The unsaturated flow of water in porous media ($\boldsymbol{u}$) is described locally by the so-called extended Darcy equation (Eq. 1) [28]. Where $K(\theta)$ is the hydraulic conductivity (with θ the volume fraction saturation), and $\boldsymbol{F}$ is the capillary force, which is identified with the negative gradient of the capillary potential ψ. Combining Eq. 1 with the continuity equation leads to the Richards equation, fundamental equation of the unsaturated flow (Eq. 2). Defining the hydraulic diffusivity function, $D = K(d\psi/d\theta)$, it becomes Eq. 3 in one dimension, with x being the distance.

$$\boldsymbol{u} = K(\theta)\boldsymbol{F} = -K(\theta)\nabla\psi \tag{1}$$

$$\frac{\partial\theta}{\partial t} = \nabla K(\theta)\nabla\psi \tag{2}$$

$$\frac{\partial\theta}{\partial t} = \frac{\partial}{\partial x}\left(D\frac{\partial\theta}{\partial x}\right) \tag{3}$$

For solving this equation, the Boltzmann transformation ($\phi = x{\cdot}t^{-0.5}$) is applied by defining $\theta = f(\phi)$. Then, Eq. 3 is written as Eq. 4. With boundary conditions $\theta = \theta_s$ at $\phi = 0$ (saturated at the surface in contact with water) and $\theta = \theta_d$ as $\phi \to \infty$ (homogeneously unsaturated semi-infinite media), the solution becomes Eq. 5.

$$-\frac{\phi}{2}\frac{d\theta}{d\phi} = \frac{d}{d\phi}D\frac{d\theta}{d\phi} \tag{4}$$

$$x(\theta, t) = \phi(\theta)t^{0.5} \tag{5}$$

This last equation shows that as the porous material absorbs, the liquid content versus distance profile advances as $t^{0.5}$ maintaining constant shape $\phi(\theta)$. When ϕ is known, the cumulative absorption i is given in Eq. 6 [29], which is the basis for the most common approach in the analysis of capillary absorption in concrete.

$$i = t^{0.5}\int_{\theta_d}^{\theta_s}\phi d\theta = S{\cdot}t^{0.5} \tag{6}$$

The common application of Eq. 6 for analysing capillary sorption in concrete faces the significant difficulty of the lack of linearity of the relationship between water uptake and $t^{0.5}$. The literature presents significant evidence of this lack of linearity [9,11,17,27,28,30–33],

Then, WSC needs to be strictly defined as $S(t)$, and a convention becomes necessary for deriving a single coefficient to describe the material. The ASTM C1585 standard [18] attempts to solve this issue by separately defining the initial and final WSCs. The initial WSC is determined as the slope of the curve during the first 6 h, while secondary WSC is computed as the slope of the same measurements between 1 and 7 days. This segmented analysis origins from significant linearity that has been reported for the initial and final testing periods. However, this is inconvenient for the assessment of durability, as two kinetic parameters are hardly able to be included in a model. Moreover, the initial coefficient is obtained over a very short test period, only involving the surface layer of concrete and unable to provide sufficient information for the modelling of capillary absorption in the bulk material [32]. The secondary coefficient is on its own not always valid, as the standard requires the verification of linearity for the segment ($R^2 > 0.98$). Then, it is important to define a single parameter, but this is hardly reachable from the relation with $t^{0.5}$. Most European standards prefer to define coefficients determined from a secant with fixed initial and final times [19,22–24,34]. This criterion is suitable for obtaining coefficients to be applied in prescriptive design only. The Argentine standard [16] instructs to calculate a single value for the parameter from a certain sector of the experiment that covers the range between 10 and 90% of the maximum water capillary absorption capacity. This procedure intends to solve the lack of linearity by imposing a conventional regression. However, the process required for repeatability ends up in a complex procedure for discarding outliers; then it impedes the value of the WSC to accurately reflect the statistical distribution of the experimental results [17,35,36]. Thus, the lack of linearity of capillary absorption with $t^{0.5}$ impedes to obtain a proper WSC, as any fabricated procedure to overcome this lack of linearity is not sustained by the physics of the process. Accordingly, an improved definition of the WSC must consider the basis for the evolution of capillary absorption in concrete with time.

Several hypotheses have been built to explain the anomaly in question, i.e. the lack of linearity between $t^{0.5}$ and water uptake of cementitious materials during sorptivity. Inhomogeneous distribution of humidity within the sample was one of the explanations [11,37], but

this thesis was refuted in Ref. [27] when samples were preconditioned for one year to allow homogeneous distribution of moisture, and they still obtained a significant deviation from the $t^{0.5}$ law. Other authors claim that the deviation from the $t^{0.5}$ law is due to the simplified balance equations that disregard a counteracting gravity term [10,31,38]. This seems unlikely as the effect of gravity must gain significance as the capillary rise approaches to the equilibrium height. At the time that the deviation starts to manifest, as early as 24 h, the capillary rise is very far from equilibrium. On this basis, other authors discard any possibility of explaining the anomaly by the effect of gravity. Martys and Ferraris [32] estimated an equilibrium capillary rise of tens of meters (whereas the usual height of a sample for the sorptivity test is usually 0.05 m). Then, the weight of the column of water in the sample is quite negligible in comparison with the capillary potential. Accordingly, Lockington and Parlange [39] estimated the times in which gravity will exert considerable influence in the order of > 10 days for mortar and more than several years for concrete. Moreover, the effect of gravity should be equally significant for all porous building materials, and data in the literature presents a specially pronounced deviation only for cementitious materials.

Nevertheless, Fries and Dreyer [40] presented an analytical solution for the capillary rise (h) restricted by gravity (Eq. 7, where W states for the Lambert W function defined by the inverse exponential function in Eq. 8, and a and b are coefficients). This solution was already presented by Washburn [41] in an implicit form (i.e. with time as a function of capillary rise), Eq. 9. Still, the achievement of the equilibrium condition takes significant time, and it is unlikely that short time tests show significant restriction by gravity in relative terms so as to make this approach necessary.

$$h(t) = \frac{a}{b}\cdot\left(1 + W\left(-e^{-1-\frac{b^2 t}{a}}\right)\right) \tag{7}$$

$$x = W(x)\cdot e^{W(x)} \tag{8}$$

$$t(h) = -\frac{h}{b} - \frac{a}{b^2}\cdot ln\left(1 - \frac{b\cdot h}{a}\right) \tag{9}$$

Therefore, our main hypothesis to explain the anomaly is the effect of the hygroscopic nature of cementitious materials. The $t^{0.5}$ approach is disregarding a significant difference between concrete and other building materials for which this approach seems sound, such as natural rocks, plaster or bricks. Cementitious materials have a particular hygroscopic nature that changes their properties according to water content. C-S-H is the main responsible agent for this difference, as the structure of this gel makes the material very affine to water and with properties that are also very much modified by moisture content. The most sound explanation seems to be the effect of swelling of the material.

4. Hygroscopicity of C-S-H in concrete

The binding of water can be clearly identified in water vapour sorption tests for very low relative humidity values, up to 40%, as water adsorbs onto the surface of pores. Here, when considering the flux of vapour through a section, the incoming amount is greater than the outcoming amount, revealing a non-steady transport process. In this case, the increase in weight of the concrete sample reveals the effect. For the case of capillary sorption, the water binding implies swelling occurring because of the hygroscopic nature of the material due to its C-S-H content. This swelling can be macroscopically measured in the specimens and microscopically detected (e.g. in samples dried for SEM observation some cracking of C-S-H is always present). Then, we can claim that this internal swelling also causes “pore refinement” regarding capillary sorption, as suggested in Ref. [32] and proven in Ref. [42,43]. The relation with $t^{0.5}$ will be naturally affected by this phenomenon. Further on, it would progressively reduce up to steady conditions. Of course reaching steady conditions is more time consuming, and whereas it might be representative of wick action with opposite surfaces for absorption and evaporation, it does not reflect the reality of wetting and drying cycles in which not enough time for achieving steady state is allowed.

This progressive limitation of transport does not occur for drying, as C-S-H contracts and allows free transport of water as vapour. Here, the smallest pores control transport, but it is not accelerated with shrinkage as evaporation in these smallest pores at the set drying temperature is the controlling process. In this case, no effect of binding can be gravimetrically detected as this binding has such a low energy that the potential driven by the drying temperature easily breaks it.

The relationship between the capillary absorption and the DR in oven supports the previous argument. The progressions of the DR and water absorption have repeatedly been modelled with the square root of time [28,32,44,45]. In Refs. [1,46], and as mentioned before also a sound relation between WSC and DR is consequently reported. This relationship is however not linear. The origin of this lack of linearity might be the lack of adjustment of the capillary absorption rate to the $t^{0.5}$ law. In fact, when the content of cement in concrete is increased, in conjunction with a reduction of the w/cm ratio, there is a relative faster decrease for the DR than for the WSC. A possible explanation for this dissimilar affectation is the fact that whereas C-S-H affects the WSC, drying occurs in an enlarging pore structure in which C-S-H shrinks and is not able to limit the removal of moisture from the sample.

According to the model proposed by Pradhan et al. [47] based on the pore size distribution of concrete, capillary absorption should be linear with the square root of time. This model considers an invariable pore structure; then a straightforward deduction is that pore network does not remain the same during water uptake.

A correlation between the water retention capacity and the resultant deformation would help to include this physical effect by assessing the delaying process in the transport mechanism. This conception means considering the deviation of the experimental curve from the proportional evolution with the square root of time as a combined effect of retention capacity and pore refinement due to swelling.

Hall et al. [48] measured expansion due to water absorption, which is also supported by observations of in-situ wetting of C-S-H under an environmental electron microscope. They linked this expansion to the interaction between incoming water and C-S-H. The expansion does not occur in tests with incoming organic liquids (for which no deformation and linear evolution with $t^{0.5}$ was obtained). This outcome is in agreement with Ref. [49], where linear organic solvent uptake with $t^{0.5}$ is reported and explained by the lack of effect of these liquids on the pore structure. It could be that testing with organic liquid would solve the problem of lack of linearity with $t^{0.5}$. However, given that capillary liquid absorption is occurring with water in the field, it is of little value to test the liquid absorption of concrete with liquids that do not produce expansion. Instead, it is necessary to consider this expansion in the model. This approach should firstly consider the interaction between water and C-S-H as the main reason for the anomalous absorption of water. Accordingly, Hall et al. [48] discard some other possible causes such as electroviscous effect, modification of the chemical composition of pore solution or the wetting contact angle. The effect of swelling reflects on the blocking of micropores and the reduction in pore connectivity. These changes are related to $t^{0.5}$ [48], which makes them fast enough so as to modify the WSC and correspond exactly to the time dependence of the anomaly for the WSC.

In consequence, there is an absorption process showing anomalous evolution with $t^{0.5}$, which is affected by expansion due to swelling that also evolves with $t^{0.5}$. An improved description of water sorptivity by cementitious materials must include this combined process during water uptake.

5. An enhanced approach considering the fourth root of time

The approach of capillary rise proportional to $t^{0.25}$ has been applied by Wagner et al. [50,51] for the case of capillary absorption of cracked

cementitious materials, but also applicable to uncracked materials. This approach is empirically based on improved fitting in comparison with the $t^{0.5}$ relation. In this regard, Parrot [11] reported an exponential coefficient of 0.32 (instead of 0.5) when testing samples dried in the air. These results also relate to the comparatively slow DR of concrete at ambient temperature. Kaufmann and Studer [27] found that after a fast initial drying, the DR evolves as $t^{0.22–0.26}$ (as a suggestive coincidence with a $t^{0.25}$ approach). During the first stage pores connected with the surface are emptied, whereas a different process takes place in the second phase that can be associated with water loss from the nanopores by linear path diffusion, with very slow progression in time.

An analogy of water transport to the theory of bilinear flow is possible. Bilinear flow occurs in hydraulically divided media when the conductivity of the crevice is finite. In this flow regime, two types of linear flow occur, one from the matrix to the crevice and one through the crevice. In the case of cementitious materials, as the primary flow would be occurring in coarser pores, being also the fastest, whereas the flow through gel pores would be the secondary flow. This secondary flow is slower and causes the noticeable deviation from the Fickian model. This analogy also relies on the fractal approach, which includes the consideration of a multiscale process taking place simultaneously at different magnitudes. This analysis is applied by Wang and Ueda [52] through a lattice network model, with a convenient adjustment. The theoretical explanation seems appropriate for the case of drying in the air, but it is, however, weak for capillary absorption, as the secondary process is unlikely to notoriously affect the first 2–3 days of water uptake, as observed in experiments.

It is also generally noted that oven drying at 100 °C or higher reduces water binding capacity, and the mass gain due to capillary absorption tends to be linear with $t^{0.5}$. The swelling of C-S-H gel modifies the microstructure of the material as it absorbs water, but at high temperatures there is a potential reduction of the activity of C-S-H due to thermal damage during drying. In this sense, it is believed that concrete with higher paste content will show more deviation from linearity with $t^{0.5}$ during absorption. However, when this is accompanied by significant reduction in the w/cm ratio, an earlier reaching of the wick phenomenon is also achieved, and the trend is again towards a linear relationship with $t^{0.5}$.

Hall et al. [30,31] dealt with the deviation from the $t^{0.5}$ law by including a negative second linear term with time (Eq. (10)) with a second coefficient, *C*. They explained this approach by a permeability term for the material Eq. (11).

$$i = S \cdot t^{0.5} - C \cdot t \tag{10}$$

$$\frac{\partial \theta}{\partial t} = \frac{\partial}{\partial x}\left(D\frac{\partial \theta}{\partial x}\right) + \frac{dK}{dx} \tag{11}$$

However, the term dK/dx referred in Ref. [53] reflects a concurrent action in the mass balance of the flow (such as water pressure due to gravity or another source during infiltration). In the case of sorptivity, this seems a forced assumption because, if the deviation from the $t^{0.5}$ relation is due to swelling, it seems unnatural to model it as a counteracting action, but it would be better included as a variation in the hydraulic diffusivity instead. Therefore and although Eq. (10) may be suitable for improved fitting to experimental data, it does not seem sustained by a precise physical description of the sorptivity phenomenon in cementitious materials. This inconsistency is notorious when we consider that the effect of gravity cannot be as high as required to cause a significant effect during capillary absorption tests on samples with 5 or 10 cm height when the capillary potential leads to a capillary rise of tens of metres. In this sense, Hall et al. [31] found more deviation from the $t^{0.5}$ law when concrete was less compacted, for the same water/cement ratio. They explain this difference by a lower capillary potential due to the presence of coarse compaction pores. However, the series of low compacted concretes showed higher sorptivity values. This higher rate of water uptake must correspond with a higher capillary potential as well. Therefore, for this case the deviation from the $t^{0.5}$ law cannot be connected with the action of gravity in the period during which capillary absorption tests take place. If the hypothesis of swelling is correct, the higher volume fraction of pores will require that the restriction needed to cause the reduction of pores will be higher. The effect of an increased compaction degree is consequently resulting in a lower porosity that results in a higher impact of internal expansion, and thus a reduced absorption rate.

6. Experimental

In concrete preparation, two different ordinary Portland cements (OPC_1 and OPC_2), one composite Portland cement (BPC), one pozzolanic Portland cement (PPC, containing approximately 35% of volcanic tuff), one ground granulated blast furnace slag (GGBFS), and one limestone powder (LP) were used as cementitious materials. Table 1 presents their properties. As aggregate, fine and coarse siliceous sands, and granitic crushed stone (GCS) with a nominal size between 6 and 20 mm were used. Water-reducing admixtures (WR) were included in all mixes to reduce the relative volume of paste.

Table 2 presents the proportions and properties of the 17 studied mixes. For OPC and w/c = 0.45, more than one batch was analysed. Duplicates and five replicates for two different paste contents were tested. One batch was prepared for the rest of mixes. The water/cement ratio of the concrete varied between 0.35 and 0.60. The mix identification is composed by a letter and subscript indicating the binder type followed by two digits, indicating the w/c ratio in %.

Cylindrical specimens of 10 cm × 20 cm in diameter and height were moulded and cured in a humid chamber (T: 23 ± 2 °C, RH > 95%) until the age of 28 days.

The gravimetric technique for the capillary absorption test was followed in accordance with Ref. [16]. Cylindrical samples (5 cm thick) cut from cast 10 cm × 20 cm cylindrical specimens were used. Five samples were tested in all cases excepting $O_1$45 batches d to h, for which 11 samples were tested in each case. The testing face of the sample was the face sawn 3 cm from the base of the specimen. The samples were waterproofed on their lateral faces with three layers of chlorinated rubber based paint, and they were oven dried with recirculation at 50 °C until they reached constant weight, i.e. when weight variation in a 24 h period was < 0.1% by weight of the sample. The drying period required, ranged between 4 and 12 days depending on the mix composition. The test itself consisted of placing the testing face of the samples in contact with water with the water level reaching 3 mm above the base of the sample. Specimens were put on thin line

Table 1
Properties of cements and supplementary cementitious materials.

Properties	BPC	OPC_1	OPC_2	PPC	GGBFS	LP
Blaine specific surface area (m^2/kg)	396	380	288	416	373	556
Retained on sieve of 75 μm (%)	3.02	1.30	2.40	2.10	< 0.01	1.60
Compressive strength						
2 d (MPa)	12.1	25.6	20.4	18.9	–	–
28 d (MPa)	40.0	45.2	44.4	41.8	–	–
Density (g/cm^3)	3.08	3.11	3.13	2.99	2.87	2.75
Chemical composition (%)						
Loss on ignition	7.96	2.14	0.80	1.20	0.87	36.01
Insoluble residue	2.97	2.50	1.40	21.65	3.40	6.65
SO_3	2.12	2.41	1.74	1.40	3.58	0.21
MgO	2.68	2.76	2.44	1.01	9.36	0.76
SiO_2	19.00	19.93	20.57	14.79	30.49	11.58
Fe_2O_3	3.49	4.00	4.30	3.09	0.29	0.68
Al_2O_3	4.38	4.30	4.22	4.77	10.68	1.82
CaO	57.44	60.38	64.55	50.32	38.17	45.82
Na_2O	0.33	0.14	0.30	0.16	1.42	0.92
K_2O	0.80	0.85	0.98	1.24	0.44	0.19
Cl^-	0.010	0.012	0.023	0.030	< 0.001	0.01
Mn_2O_3	n/d	n/d	n/d	n/d	0.50	0.16

Table 2
Proportioning and properties of concretes.

Materials (kg/m^3)	$O_1$35	$O_2$35	$O_1$40	$O_2$40	$O_1$45		$O_1$50	$O_2$60	S40	L40	SL40	C40	C45	C60	P35	P40	P60
					a,b	c,d,e,f,g,h											
Water	133	140	140	140	144	153	150	164	140	140	140	140	144	168	140	182	170
OPC_1	380	–	350	–	320	345	300	–	227	262	227	–	–	–	–	–	–
OPC_2	–	400	–	350	–	–	–	274	–	–	–	–	–	–	–	–	–
BPC	–	–	–	–	–	–	–	–	–	–	–	350	320	280	–	–	–
PPC	–	–	–	–	–	–	–	–	–	–	–	–	–	–	400	442	290
GGBFS	–	–	–	–	–	–	–	–	123	–	88	–	–	–	–	–	–
LP	–	–	–	–	–	–	–	–	–	88	35	–	–	–	–	–	–
Fine sand	189	188	190	190	193	183	193	242	190	190	190	190	193	187	182	100	116
Coarse sand	749	739	754	754	766	724	767	726	755	755	755	754	766	742	713	700	813
GCS 6–20	980	979	980	980	980	1000	980	959	980	980	980	980	980	980	979	981	992
WR 1/2/3/4 (l)	6.2	6.1	5.9	5.25	6–5.71	1.8	3.6	2.74	4.9	4.2	4.1	5.25	4.80	2.80	7.4	–	–
Air (%)	3.0	4.2	3.1	3.0	3.0–4.0	4.5–5.5	3.1	3.0	3.2	3.5	3.4	3.0	4.0	3.0	3.2	1.7	2.2
Properties																	
Unit weight (kg/m^3)	2404	2392	2417	2404	2392–2404	2351–2385	2404	2354	2392	2354	2385	2392	2354	2354	2417	2404	2380
Slump (cm)	8.0	11.0	10.0	8.0	6.0–8.0	5.5–12.0	6.0	10.0	10.0	9.0	7.0	10.0	7.0	10.0	16.0	4.5	3.0
Bleeding (%)	< 0.01	< 0.01	< 0.01	n/d	3.03-n/d	3–6.9	0.40	n/d	< 0.01	< 0.01	< 0.01	n/d	n/d	n/d	< 0.01	n/d	n/d
Absorp. 24 h (%)	3.37	2.50	3.64	3.20	n/d	n/d	n/d	n/d	4.58	3.77	4.55	3.95	n/d	n/d	2.84	5.00	5.15
Porosity (%)	8.02	6.02	8.68	7.66	n/d	n/d	n/d	n/d	10.60	8.78	10.49	9.12	n/d	n/d	6.81	11.55	11.75
Compressive strength																	
7 d	54.2 (2.9)	53.2 (1.7)	47.6 (0.5)	n/d	37.8 (1.1) n/d	36.1 (0.4) 34.5 (1.9) 30.7 (2.9) 34.6 (0.6) 37.4 (1.5) 35.4 (0.6)	37.4 (1.4)	n/d	47.6 (0.5)	37.4 (2.3)	43.6 (1.7)	n/d	n/d	n/d	52.4 (0.30)	n/d	n/d
28 d	60.2 (3.0)	58.2 (3.4)	53.8 (2.5)	49.2 (1.0)	45.6 (1.3) 44.3 (1.7)	42.5 (0.4) 40.6 (0.4) 37.5 (1.3) 39.3 (1.1) 42.1 (0.8) 42.8 (1.3)	45.3 (1.1)	36.7 (1.0)	52.6 (1.3)	42.2 (0.5)	51.1 (1.5)	40.3 (2.6)	29.1 (0.8)	25.3 (0.8)	57.4 (3.0)	31.5 (0.8)	23.0 (1.3)
90 d	n/d	67.7 (0.7)	n/d	n/d	n/d	n/d	n/d	n/d	53.1 (2.0)	47.3 (5.0)	60.8 (4.0)	n/d	n/d	n/d	65.0 (8.1)	44.5 (2.0)	30.5 (1.7)

supports to separate them from the base of the container and the face put in full contact with water. The weight increments at regular intervals (0.5, 1, 2, 3, 4, 5, 6, 24 h, and from there every 24 h) were recorded, until the increase was < 0.1% of the actual weight of the sample in a 24 h period.

7. Results

Fig. 1 shows the results of capillary suction for all series. Error bars represent maximum and minimum values, and ± standard deviation on the mean. For better clarity, only variations of $O_1$45c and $O_1$45e are

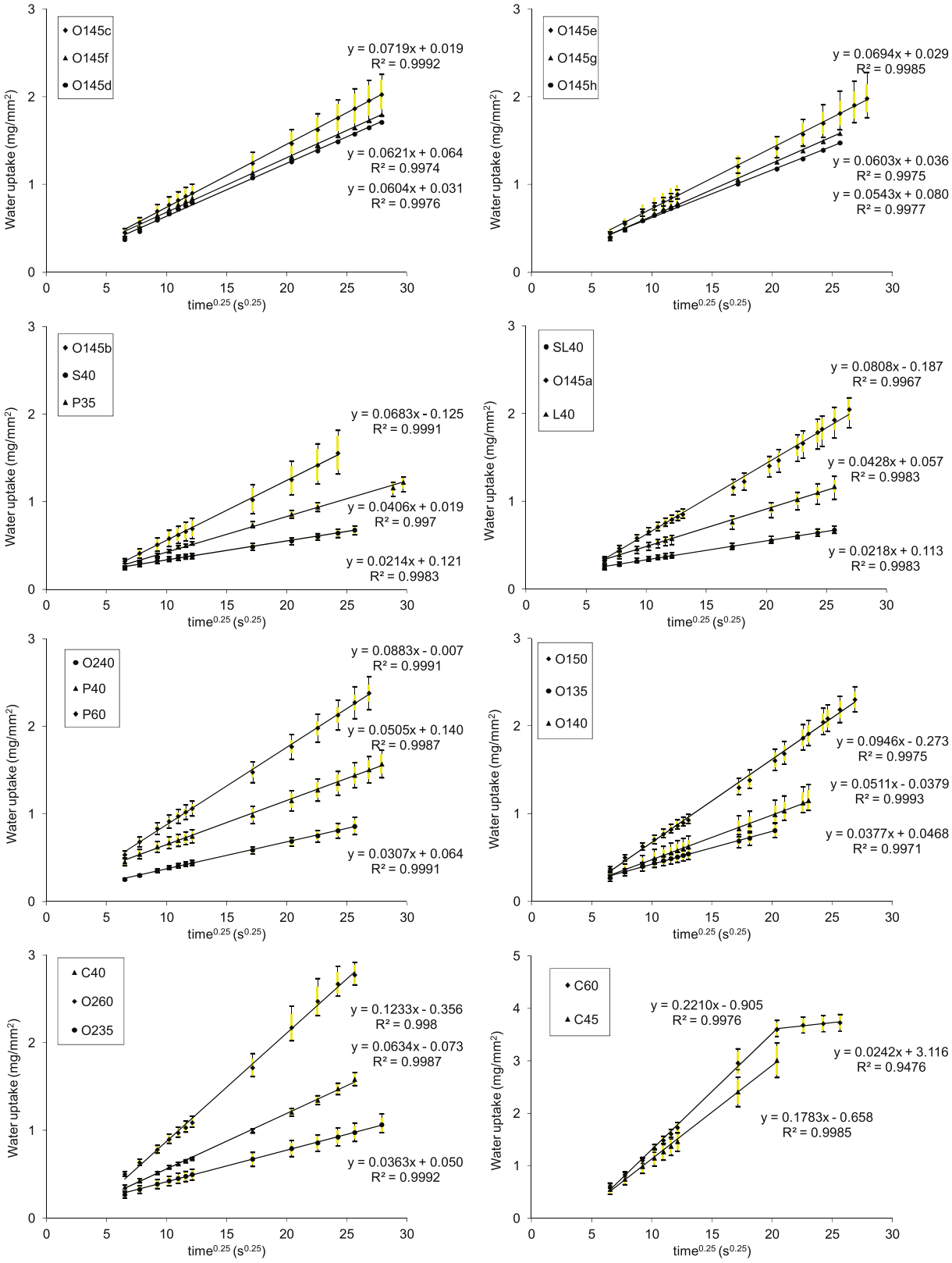

Fig. 1. Results for capillary absorption of all studied concrete mixes.

presented in the first two subfigures, which are similar to the variations obtained for the other four series in these two subfigures for which only mean values are presented. Linear regression was applied to the results for obtaining capillary absorption rates, and the progression of water uptake shows utmost linearity with $t^{0.25}$. The coefficient of determination R^2 between water absorption and $t^{0.25}$ is higher than 0.9945 for all series.

The interceptions of the equations were not forced to zero as experimentally it is assumed that the small submersion needed to assure contact between the samples and water (3 ± 1 mm) minimally affects this value, which can also be affected by the condition of the surface of the sample. There is no clear correlation between the values for this interception coefficient and the properties of the mixes. The only interpretation of this non-zero coefficient seems to be linked to the set-up of the experiment and not with the properties of the material itself. Thus, the only parameter to be analysed is the slope of the curves.

There is a significantly improved description of the phenomenon with the $t^{0.25}$ approach in comparison with the correlation with $t^{0.5}$ (i.e., when the capillary absorption rate is computed in accordance with the Argentine standard [16]), as shown in Fig. 2. Regarding the accuracy of each approach, it is important to analyse the different reasons for which there is no interception at zero in the axis in each case. Whereas the non-zero intersection for $t^{0.25}$ is of small significance and can be explained by the experimental setup, the non-zero intersection for $t^{0.5}$ is vital for a good empirical correlation but cannot be physically justified. Whereas the significant linearity with $t^{0.25}$ allows attributing most of the deviation to the experimental setup, for the case of the $t^{0.5}$ approach, in addition to the effect of the setup, an artificially increased deviation from the origin of the regressed line is provoked when the curvature of the progression is approached by a straight line. Here, considering later data with an increasingly decreased slope with time augments the value for the intersection at time = 0. As a consequence, the comparison is naturally much more favourable for the $t^{0.25}$ when the intersection with the axis is forced to zero, with R^2 decreasing in approximately a 10% for the worst case. A zero intersection cannot be even considered for the $t^{0.5}$ approach as R^2 descends to values near 0.5 given that the line intersects the experimental curve in a single point. Then, even when the comparison between the goodness of fit for each approach is favourable to the $t^{0.25}$ approach, this is not its strongest point. The $t^{0.25}$ does not require any artificial convention for determining a water sorptivity coefficient.

The C60 series presents a particular case. Here, the capillary rise reached the top face of the samples, but the increase in weight

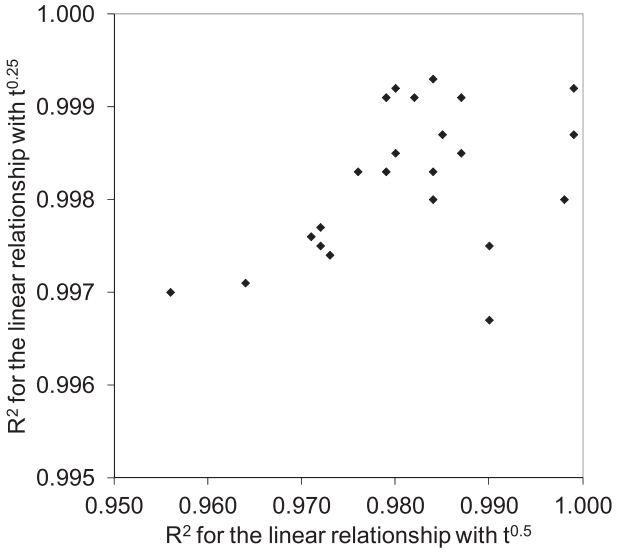

Fig. 2. Comparison between coefficients of correlation for linear relationships with $t^{0.5}$ versus $t^{0.25}$.

continued after this point at a much slower rate. This second stage is not the object of this study, but the particular coincidence that the progression of weight increase continued to be proportional to $t^{0.25}$ even when capillary absorption terminated requires further research. More knowledge in this sense will be particularly useful for long-term capillary absorption processes beyond the period in which the capillary absorption tests take place.

8. Discussion

Considering the excellent fitting of our experimental results with the $t^{0.25}$ approach, we also intend to confirm this good fitting with data in the literature.

Fig. 3(a,b,c) presents data in Ref. [54] for capillary absorption tests on mortar samples. The authors compared four increasingly severe types of preconditioning: drying in 85, 65 and 50% RH environment, and oven drying at 105 °C. The data for series w/c = 0.45, dried at 85% (Fig. 3c) and w/c = 0.40, dried at 85% (Fig. 3b) are the same. This information was taken from the reference and is probably a reporting mistake. Two absorption stages progressing at different rate are distinguishable in the original data. Only the first stage is presented and analysed here (as well as later for data in Fig. 6), as the second stage develops after the capillary rise has reached the total height of the samples, and consequently connected with a process distinct from capillary absorption (probably explained by the fractal theory). Fig. 3 shows the good fit of data to the $t^{0.25}$ approach. It is important to mention that the first considered value corresponds to 24 h of exposure, and thus the first hours of capillary absorption are not included. Noteworthy, the samples show a faster capillary absorption rate during short-term tests when they were subjected to more intensive drying. These results can be considered an indication of a reversibility of the process, as the capillary rise fully develops from the capillary potential with no delaying effect of swelling. Added to the damage that oven drying can cause in the microstructure, drying at 105 °C can also damage the structure of C-S-H. Despite the damage seems not severe enough so as to limit completely the ability of C-S-H to retain water, it tends to increase linearity between water uptake and $t^{0.5}$.

Dias [9] also suggested a duration of the first stage in association with concrete quality. The longer the first stage, the lower the WSC of concrete. This observation is also possible in Fig. 3, where lesser severity in drying resulted in longer linear periods with $t^{0.25}$. This correlation can then be linked to the preconditioning of samples, where less intense drying caused longer periods for the first stage in agreement with an increasingly closed microstructure. This tortuosity effect may also increase with lower w/cm ratios or the use of supplementary cementitious materials.

Similar excellent fitting with $t^{0.25}$ from data in Refs. [8,5,32,55,28] is presented in Figs. 4, 5, 6, 7, and 8, respectively. Only one stage is observed in these cases as the testing period is short. Fig. 4 presents results of high strength concrete with and without air entrainment, and self-consolidating concrete. Fig. 5 presents results from concrete samples at ages of 28 and > 400 days, with w/cm ratios of 0.68, 0.38, and 0.28, and concrete containing 30% fly ash and 10% silica fume. Here, the results for PO mixes with w/c 0.38 and 0.28 seem to be a reporting mistake. Figs. 6, 7 and 8 present samples made with mortar. They respectively show: standard and high performance OPC mortar mixes, standard 0.5:1:3 water:cement:sand ratio mortar with and without chemical admixtures (plasticizer and air entraining agent), and 0.4:1:2 water:cement:sand ratio standard mortar. This set of data includes a wide variety of mixes, and they all fairly follow the $t^{0.25}$ approach.

9. Definition of a new theoretical model

From the literature review, three potential causes are the most sustained explanations of the anomalous capillary absorption by cementitious materials for tests conducted for short term (up to 250 h).

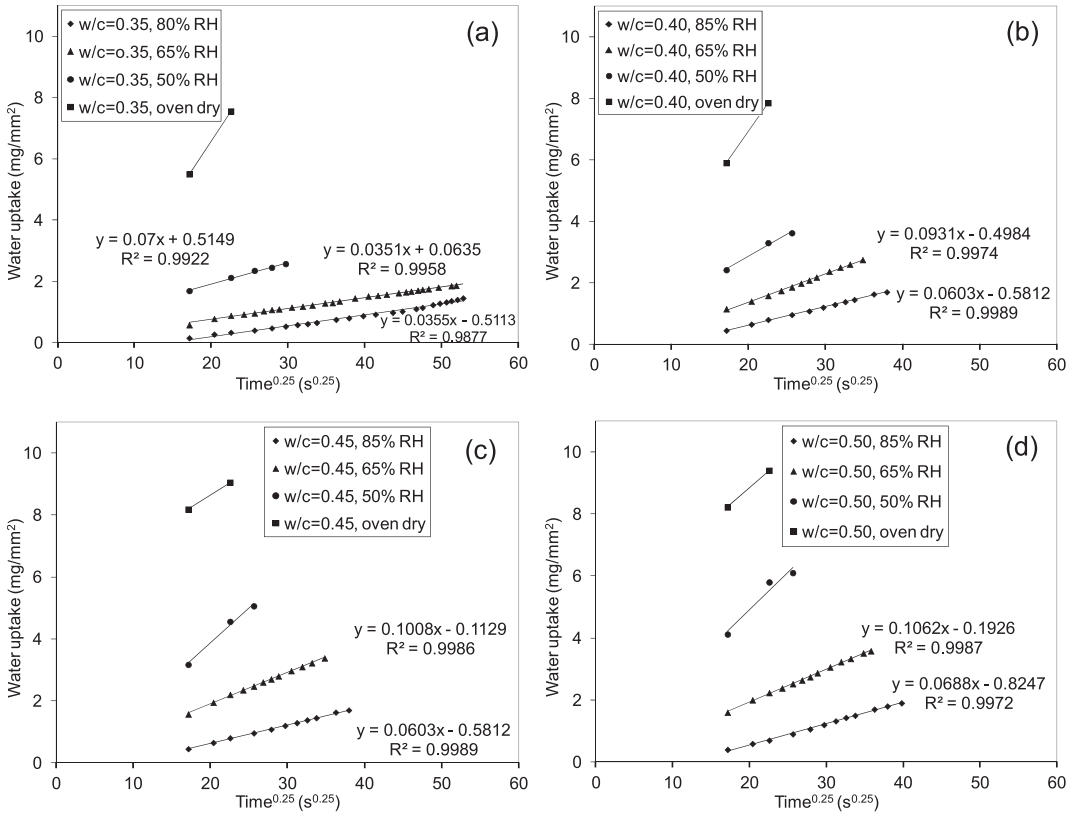

Fig. 3. Fitting of data from mortars in Ref. [54], a) w/c = 0.35, b) 0.40, c) 0.45 and d) 0.50, with four increasingly severe drying preconditioning procedures.

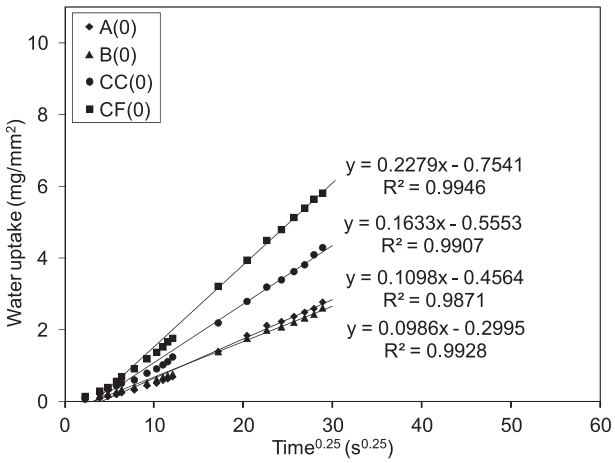

Fig. 4. Fitting of data for concrete mixes from Ref. [8]. A(0) = high strength concrete; B (0) = high strength concrete with air entrainment; CC(0) = self-consolidating concrete tested on cast surface; CF(0) = self-consolidating concrete tested on finished surface.

These are the effect of gravity, the pore structure requiring a description by the fractal theory, and the swelling of the material. At first the most convincing is swelling, as gravity and one-dimensional transport seem to be unsuitable to explain the anomaly in such a short measurement time.

Regarding the restriction by gravity, Fig. 9 shows the fitting of data from $O_1$45d to Eqs. 7 and 9. Only this series is presented as an example since all other series show similar results. It is noticed that some limitations arise from the interval in real numbers for which the W function is defined ($x \geq -1/e$), and some differences in the modelled time progression of water uptake come up from the application of Eq. 7 or Eq. 9. These correlations to experimental data are not as good as the $t^{0.25}$ approach. Whereas the explicit solution fits better for the initial measurements and reveals a fictitious stabilisation of the capillary rise at the end of the test, the regression to the implicit solution fits better to the latest measurements in detriment of the correlation with the initial period. This last case would be the more realistic of the two, but it is notorious that it fails when attempting to describe the progressive reduction during the first hours of water uptake. As suspected, data do not support gravity as the main reason for the anomaly.

The consideration of a multidimensional model for the pore structure requires the transport mechanism to be analysed as a non-Fickian process. A non-Fickian model has been formulated for diffusion in disordered media [56], considering the fractal theory. They can describe the broad pore size distribution in concrete, especially the scale differences between capillary and C-S-H pores. This model has been experimentally supported by results from different microporous materials also showing anomalous transport phenomena (diffusion [57], dispersion [58], infiltration [59]). Here, Eq. (1) is substituted by a more general form (Eq. 12), where n is a real number. Combining Eq. 12 with the continuity equation leads to Eq. 13, for which Eq. 4 is a special case for $n = 1$.

$$\boldsymbol{u} = D(\theta)\left(\frac{d\theta}{dx}\right)^n \tag{12}$$

$$\frac{\partial\theta}{\partial t} = \frac{\partial}{\partial x}\left(D(\theta)\left(\frac{\partial\theta}{\partial x}\right)^n\right) \tag{13}$$

In a similar manner to the resolution of Eq. 1, a transformation ($\phi^* = x{\cdot}t^{-\alpha}$) can be applied by defining $\theta = f(\phi^*)$. Then, Eq. 13 reduces to Eq. 14, provided that $t = t^{\alpha(n-1)}$, i.e. $n = 1/\alpha - 1$. With boundary conditions $\theta = \theta_s$ at $\phi^* = 0$ and $\theta = \theta_d$ as $\phi^* \to \infty$, the solution becomes Eq. 15.

$$\frac{d^2\theta}{d\phi^{*2}} + \frac{1}{n{\cdot}D(\theta)}\frac{dD(\theta)}{d\theta}\left(\frac{d\theta}{d\phi^*}\right)^2 + \frac{\alpha}{n}\frac{\phi^*}{D(\theta)}\left(\frac{d\theta}{d\phi^*}\right)^{-n+2} = 0 \tag{14}$$

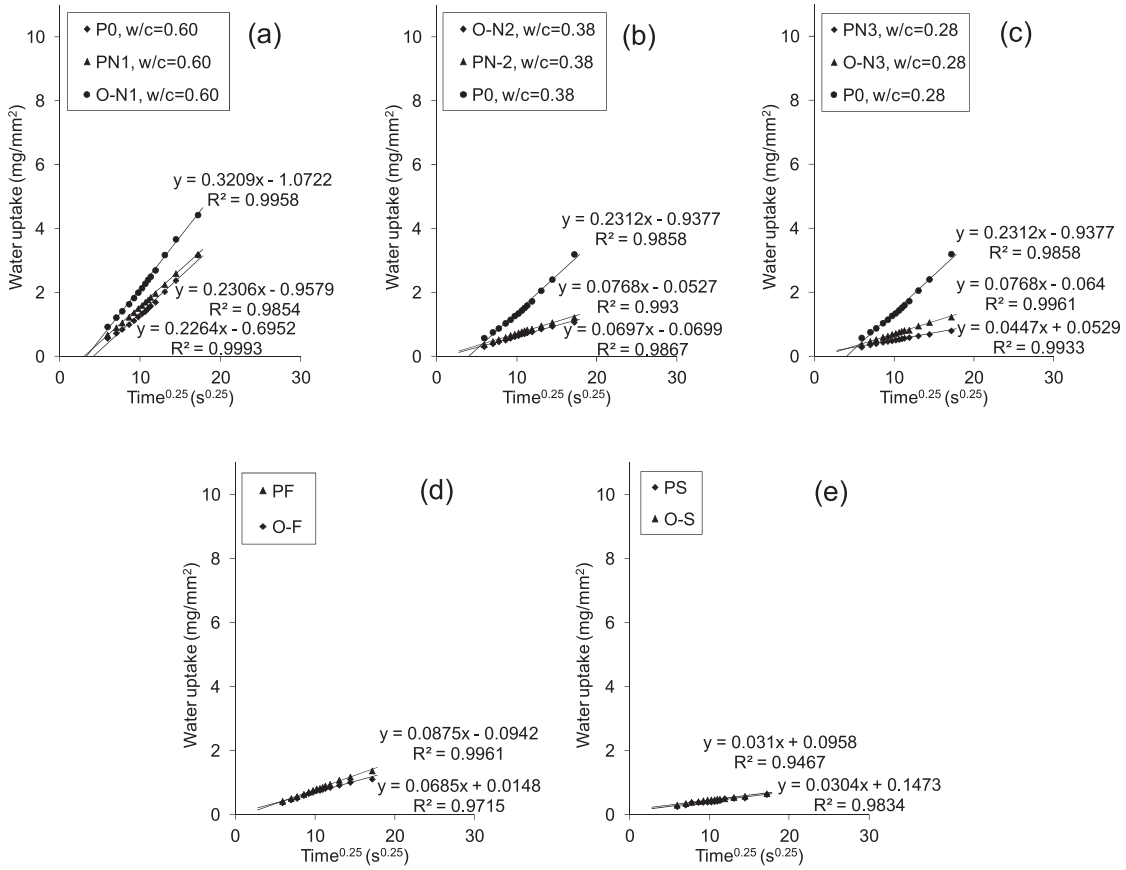

Fig. 5. Fitting of data for concrete mixes from Ref. [5]. Nomenclature: P = concrete with pure OPC, O = concrete with > 400 days of age, N = concrete with 28 days, ON = composite samples of two layers of 400-day concrete and 28-day concrete, F = 30% fly ash concrete, S = 10% silica fume concrete. a) OPC, w/c = 0.60, b) OPC, w/c = 0.38, c) OPC, w/c = 0.28, d) OPC + fly ash, w/cm = 0.38, e) OPC + silica fume, w/cm = 0.38.

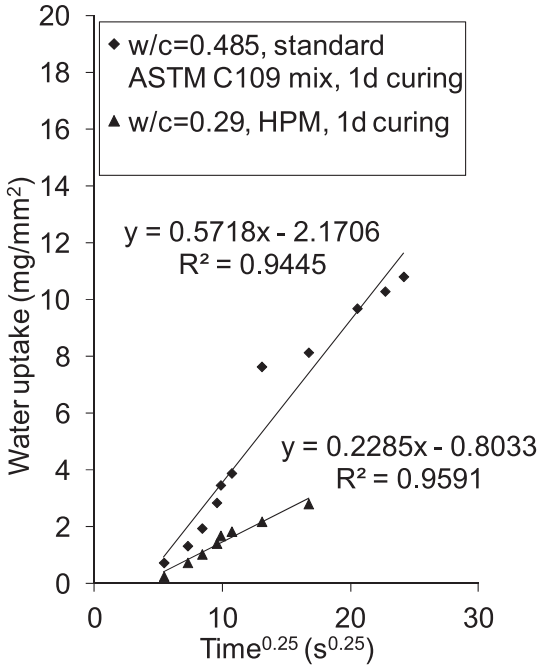

Fig. 6. Fitting of data for mortar mixes from Ref. [32]. HPM = high performance mortar.

$$x(\theta t) = \phi^*(\theta) t^{\alpha} \tag{15}$$

The constant shape $\phi^*(\theta)$ of the liquid content advancing profile maintains, and it advances as t^{α}. With a known ϕ^*, the cumulative absorption i is therefore given by Eq. 16, which is the basis for the non-Fickian approach in the analysis of capillary absorption in concrete. This equation has been already empirically applied by Kaufman and Studer [27] to capillary absorption in concrete.

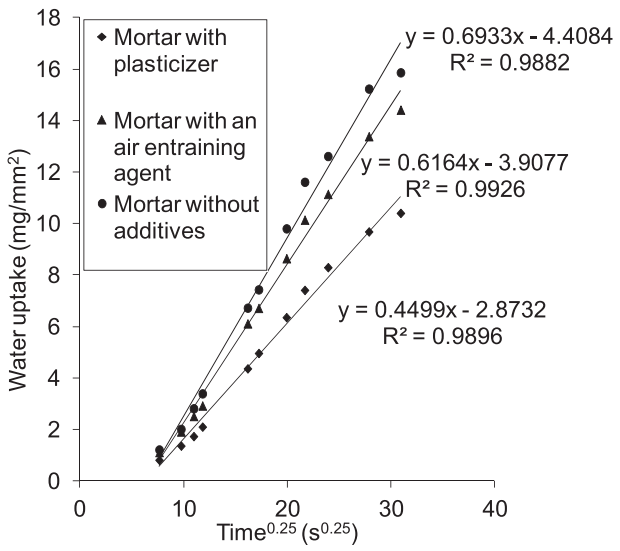

Fig. 7. Fitting of data from Ref. [55]. Mortar mixes with and without chemical admixtures, w/c = 0.50.

$$i = t^{\alpha} \int_{\theta_d}^{\theta_s} \phi^* d\theta = S^* \cdot t^{\alpha} \tag{16}$$

If $\alpha = 0.25$, then $n = 3$, implying that the flux of water due to

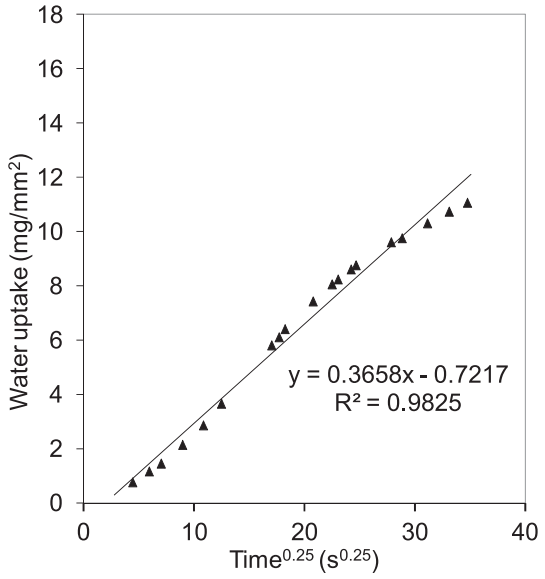

Fig. 8. Fitting of data from Fig. 4.20 in Ref. [28]. OPC mortar with 0.4:1:2 water:-cement:sand ratio.

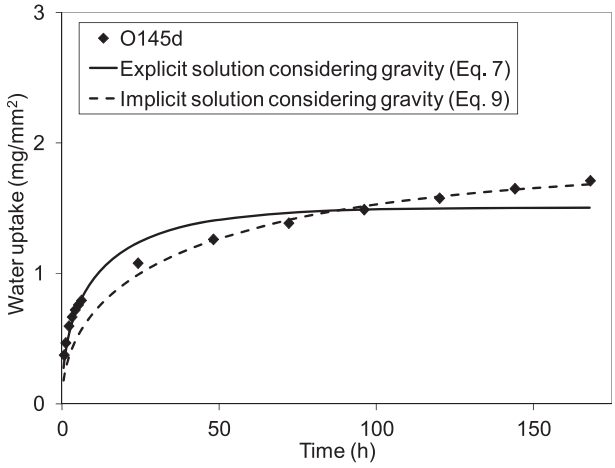

Fig. 9. Correlation of experimental data from O145d and Eqs. 7 and 9.

capillary absorption is proportional to the cube of the moisture gradient. This approach seems to correspond well with the experimental results.

However, if the transport process is modelled using a double-diffusive mechanism in capillaries and gel pores, the flow in these two phases would show a significant time scale difference, leading to the lack of linearity with $t^{0.5}$. This being the case, it is assumed that the flow in gel pores is purely one-dimensional and much slower than the process of capillary absorption. The model is equivalent to considering a homogenous body of C-S-H interrupted by capillary pores as discontinuities. This analysis can be applied to both bidimensional and bilinear models, being the same proposal to that by Rucker-Gramm and Beddoe [60]. Given the anomalous capillary absorption for the short-term experiments, it seems unlikely that one-dimensional diffusion is the reason for the deviation of capillary absorption from the linear relationship with $t^{0.5}$ during the first days of exposure. The secondary transport process would be too slow so as to show significant effect during this initial period. It could be thought that the influence of very small pores reveals at later exposure periods, or when the capillary rise has reached the total height of the sample, but not around only 6 h after exposure.

Indeed, both water uptake and capillary rise present a deviation from linearity with $t^{0.5}$. In other words, for short-term tests, there is no detection of transport through micropores by a weight increase that does not replicate in the capillary rise. When the increase in weight is faced against the measurement of the wet front height, as indicated by data reported in Ref. [55] (Fig. 10), a fair capillary absorption process arises as the height of the wet front is proportional to the water uptake. Similar results are reported in Ref. [49], for which the evolution is also linear with $t^{0.25}$ for this data set (Fig. 10). The capillary rise represents all transport of water, and the only explanation for deviation from the $t^{0.5}$ law seems to be a reduction in the hydraulic diffusivity, with no experimental indication of a secondary transport process.

For short-term tests (at least up to 250 h, depending on the properties of the mix), the deceleration of capillary absorption seems to be a result of swelling. The transport of water can only be connected with the capillary absorption process with variable hydraulic diffusivity.

With this in mind, there is no deviation of the water flux-gradient proportionality, but a variation on the hydraulic diffusivity of the material instead. This approach has been applied in Refs. [39,61] for porous materials after derivation of the theoretical basis. They propose the consideration of Richard's equation with a time-dependent diffusivity (Eq. 17). Diffusivity is considered to be a separable function of θ and t, as in Eq. 18, sustained by the lack of evidence of a variation of the

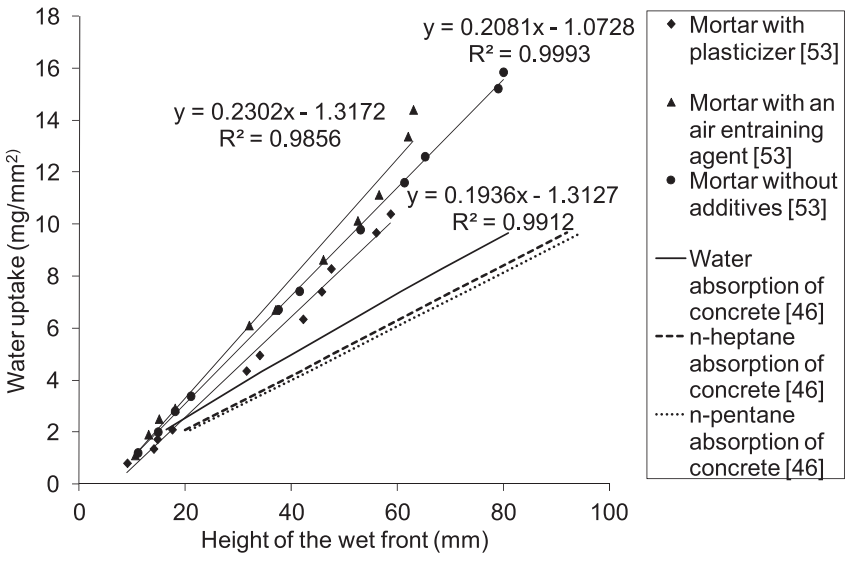

Fig. 10. Relationship between the water uptake and the height of the wet front. From Refs. [49,55].

shape of the front with time (even with the consideration of a non-Fickian model). With the substitution of variables in Eq. 19, Eq. 17 can be rearranged as Eq. 20, with boundary conditions $\theta = \theta_s$ at $x = 0$, $\tau > 0$, and $\theta = \theta_d$ at $x > 0$, $\tau = 0$, and $\theta = \theta_d$ at $x \to \infty$, $\tau > 0$. After Boltzmann transformation, this brings an analogous solution to Eq. 7, as Eq. 21.

$$\frac{\partial \theta}{\partial t} = \frac{\partial}{\partial x}\left(D(\theta t)\cdot\left(\frac{\partial \theta}{\partial x}\right)\right) \tag{17}$$

$$D(\theta t) = \gamma(t)\cdot\delta(\theta) \tag{18}$$

$$\tau = \int_0^t \gamma(a)da \tag{19}$$

$$\frac{\partial \theta}{\partial \tau} = \frac{\partial}{\partial x}\left(D(\theta)\cdot\left(\frac{\partial \theta}{\partial x}\right)\right) \tag{20}$$

$$i = S^{*}\cdot\tau^{0.5} \tag{21}$$

Now, the problem reduces to the definition of γ for accounting the evolution of hydraulic diffusivity. This influence is, however, not easy to predict.

In mathematical terms, this model for the short-term water uptake requires the consideration of the evolution of pore connectivity and tortuosity with time as a consequence of swelling. The model of Powers for the proportional relationship between permeability and the cube of porosity [62] is well-known worldwide. However, this is considering constant conditions for the fluid passing through the material, meaning no affectation of the pore network during the transport process as in the case of capillary absorption. Expansion due to water uptake of cementitious materials presented in Hall et al. [48] progresses linearly with $t^{0.5}$. It seems evident that expansion was occurring only in the zone over which the wet front had progressed. Then, specific expansion should only be considered in terms of the height of capillary rise. We consequently derive that expansion and water rise progress at the same rate, and the effect of swelling on the water uptake rate should account for this.

Volumetric expansion due to swelling can be obtained considering the material as isotropic. Accordingly, the volumetric expansion also progresses linearly with $t^{0.5}$. A subsequent connection between the documented external expansion and the modification of the pore network is needed.

Swelling can cause not only external expansion but also internal expansion due to internal restrictions of the material. Thomas and Jennings [63] showed that if the degree of restraining (with values 0 = no restraint and 1 = full restraint) is higher than a value numerically equivalent to the initial volume fraction of pores, porosity will reduce due to expansion. This very low restriction required is certainly achieved during water uptake, leading to pore shrinkage caused by swelling after water absorption. In this sense, it should be noted that the higher w/cm ratio would indicate less restriction to deformation, which means less reduction of porosity due to swelling and a consequent increased linearity of water uptake progression with $t^{0.5}$. Contrary to the simple assumption that a higher cement content will cause higher sensitivity of the material to water absorption, it is the porosity of the material that will define the sensitivity. From the relationship between pores, deforming phase and non-deforming phase, the deformation coefficient for pores will be proportional to the deformation coefficient of the material. Then, the pore network will evolve with time in proportion to the evolution of the solid phase. From this, we considered that the evolution of hydraulic diffusivity will be proportionally affected by swelling, and consequently evolving as $t^{0.5}$ with water uptake, following the development of external expansion.

This correlation between hydraulic diffusivity and external expansion seems a consistent approach, and experimental evidence on it seems a very useful information to collect in future research.

Based on the exponential evolution for hydraulic conductivity, after considering swelling with internal restriction, Eq. 19 becomes $\tau = t^{0.5}$, meaning γ as Eq. 22. Finally, Eq. 21 can be expressed in terms of time as Eq. 23, giving theoretical support to the empiric correlation of experimental data. Lockington et al. [39] presented different values for the exponential coefficient that relates t and τ, based on the best fit to experimental data from Ref. [64]. These values are near 0.5 for all cases, and, based on the evolution of expansion, a single value equal to 0.5 seems a consistent approach that leads to Eq. 23.

$$\gamma(t) = \frac{t^{-0.5}}{2} \tag{22}$$

$$i = S^{*}\cdot t^{0.25} \tag{23}$$

The anomalous capillary absorption in cementitious materials, non-linear with $t^{0.5}$, has been documented and discussed for a long time. A possible explanation for continuing using the $t^{0.5}$ could be the lack of a theory that fully explains this deviation from the conventional model for capillary absorption. In this paper, we presented excellent fit of the progression of water uptake with $t^{0.25}$, and we analysed the possible causes of this strong empirical evidence. Accordingly, the theoretical basis of the new $t^{0.25}$ approach has been presented in this paper considering the effect of swelling and its evolution with time. As swelling of C-S-H affects the pore size distribution of pre-dried cementitious materials when they get in contact with water, it causes a reduction in hydraulic diffusivity due to internal restrictions to the deformation of the material. This is therefore a sound explanation for the anomalous capillary absorption.

As a result, the capillary absorption rate determined as the slope of the relation between the water uptake and the fourth root of time allows representing the full range of experimental data for tests conducted at least up to 250 h provided that further capillary rise is feasible.

10. Conclusions

Experimental results for the capillary absorption of cementitious materials presented in this paper and the literature, challenge the usual approach of water uptake in proportion to the square root of time. This anomaly has been known for some time already and awkwardly assessed by standards for the computation of water sorptivity coefficient. A thorough consideration of this issue requires abandoning the $t^{0.5}$ approach for capillary absorption in cementitious materials. The capillary absorption rate is better defined as the ratio between the water uptake and the fourth root of time, and this finds fundament in the hygroscopicity of cementitious materials and swelling caused by the interaction with water. Unlike other building materials, the content of C-S-H in cementitious materials induces a deviation from the relationship with $t^{0.5}$, which seems a more sound explanation than the influence of gravity or bi-dimensional transport. Additional experimental support is required to clarify this aspect.

The preconditioning and in particular the drying temperature play a significant role in this matter, as the C-S-H activity can be reduced by drying at a temperature of 105 °C. With high drying temperature, we can expect improved linearity with $t^{0.5}$. This apparent improvement is, however, in detriment of a realistic assessment of the material in service conditions.

The definition of the WSC as the rate of water uptake progressing over the fourth root of time solves the usual problem of lack of linearity observed with the square root of time. This $t^{0.25}$ approach is effectively supported by our own and other authors' experimental data for tests conducted for up to 10 days or the time required to cover the total height of the samples. Capillary absorption with varying hydraulic diffusivity dominates this first stage of transport. During the first days of exposure, the delaying effect of water retention and swelling on the flow of water due to capillary absorption finds an appropriate model in the linear evolution of water uptake with $t^{0.25}$. This delaying process is fast enough to show significant influence on the water flow rate due to

capillary absorption. In this manner, a single parameter is obtained allowing a significant simplification and universalisation for the analysis of results from the capillary absorption test.

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